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Technical Report ARAED-TR-88017

PRESSURE/TEMPERATURE SENSITIVE INORGANIC PHOSPHORS

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September 1988

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SECURITY CLASSIFICATION OF THIS PAGE

REPORT DOCUMENTATION PAGE

1a. REPORT SECURITY CLASSIFICATION UNCLASSIFIED	1b. RESTRICTIVE MARKINGS
2a. SECURITY CLASSIFICATION AUTHORITY	3. DISTRIBUTION/AVAILABILITY OF REPORT Approved for public release; distribution is unlimited.
2b. DECLASSIFICATION/DOWNGRADING SCHEDULE	
4. PERFORMING ORGANIZATION REPORT NUMBER Technical Report ARAED-TR-88017	5. MONITORING ORGANIZATION REPORT NUMBER)
6a. NAME OF PERFORMING ORGANIZATION ARDEC, AED Energetics and Warheads Div 6b. OFFICE SYMBOL SMCAR-AEE	7a. NAME OF MONITORING ORGANIZATION ARDEC, AED Energetics and Warheads Div
6c. ADDRESS (CITY, STATE, AND ZIP CODE) Picatinny Arsenal, NJ 07806-5000 (cont)	7b. ADDRESS (CITY, STATE, AND ZIP CODE) Picatinny Arsenal, NJ 07806-5000
8a. NAME OF FUNDING/SPONSORING ORGANIZATION ARDEC, IMD STINFO Br 8b. OFFICE SYMBOL SMCAR-IMI-I	9. PROCUREMENT INSTRUMENT IDENTIFICATION NUMBER
8c. ADDRESS (CITY, STATE, AND ZIP CODE) Picatinny Arsenal, NJ 07806-5000	10. SOURCE OF FUNDING NUMBERS PROGRAM ELEMENT NO. / PROJECT NO. / TASK NO. / WORK UNIT ACCESSION NO.

11. TITLE (INCLUDE SECURITY CLASSIFICATION)
PRESSURE/TEMPERATURE SENSITIVE INORGANIC PHOSPHORS

12. PERSONAL AUTHOR(S) William O. Seals, ARDEC; Henry W. Offen, University of California; W. Dale Turley and Henry M. Borella, EG&G Energy Measurements Inc.

13a. TYPE OF REPORT	13b. TIME COVERED	14. DATE OF REPORT (YEAR, MONTH, DAY)	15. PAGE COUNT
Technical	FROM Sep 87 TO Feb 88	September 1988	26

16. SUPPLEMENTARY NOTATION

17. COSATI CODES			18. SUBJECT TERMS (CONTINUE ON REVERSE IF NECESSARY AND IDENTIFY BY BLOCK NUMBER)
FIELD	GROUP	SUB-GROUP	Inorganic phosphors, Fluorescence decay, Kistler gauges, Lanthinum oxy-sulfide, Pressure transducer, Self-propelled howitzer, Yttrium oxy-sulfide, Hydraulic pressures

19. ABSTRACT (CONTINUE ON REVERSE IF NECESSARY AND IDENTIFY BY BLOCK NUMBER)

A class of inorganic phosphors, such as rare earth doped lanthinum oxy-sulfide and Yttrium oxy-sulfide, show spectral emission characteristics that are strongly pressure and temperature dependent. The relative intensity of the emission lines and the fluorescence decay time have indicated strong pressure dependence in the range of several kilobars. This property suggests that these inorganic phosphors could be applied as remotely operated pressure transducers. The intensity of the emissions has also been observed to show strong dependency upon temperature.

Kistler gauges are presently used to monitor pressures. Prior vulnerability studies indicated that hydraulic pressures in excess of 100,000 psi were experienced when one gallon of stored liquid propellant was impacted by a shaped charge. These pressures exceed the upper limit of the Kistler gauges. In field use, large quantities will be housed in an armored or self-propelled howitzer. It is critical to measure the pressures developed in these larger quantities of stored propellant when impacted by a battlefield threat. This report discusses the potential for using organic phosphors to measure high pressures and temperatures.

20. DISTRIBUTION/AVAILABILITY OF ABSTRACT	21. ABSTRACT SECURITY CLASSIFICATION
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INTRODUCTION

In the vulnerability assessment of liquid propellants, the Ballistic Research Laboratory encountered a problem in monitoring the hydraulic pressures produced within the liquid propellant when impacted by a shaped charge. Kistler gauges were initally used to measure the pressures generated. Unfortunately, the pressures experienced in a one-gallon container of liquid propellant under shock loading exceeded the limitation of the Kistler gauges. These pressures were in excess of 100,000 psi. Normally, liquid propellant will be stored in larger quantities. It is critical to know the pressure generated in these larger stored quantities of liquid propellant in order that safety design criteria for storage containers can be obtained. Another concern is the safety of the crew members of an army vehicle from the high hydraulic pressures produced.

The Army Research, Development and Engineering Center initiated a program to ascertain the feasibility of using pressure and temperature-sensitive inorganic phosphors to remotely monitor these parameters.

BACKGROUND

A class of inorganic phosphors that include rare earth doped lanthanum oxy-sulfide (La_2O_2S) and yttrum oxy-sulfide (Y_2O_2S) have demonstrated spectral emission characteristics that are strongly pressure-dependent (ref 1). These emission spectra are extremely complicated. More than 200 emission lines can be obtained. To be able to understand these emissions, a few representative lines are selected for study. To insure that the pressure affected the crystal states equally, the emission lines originating in the upper ^5D_i levels were monitored. The results from these studies demonstrated that all the emission lines originating from the same ^5D_i term displayed the same pressure dependence. The emission lines selected from other ^5D_i level emissions assured that the pressure dependency for a given emission line represented the effects at only one level. As the magnitude of the pressure was increased, the phosphorescence intensity increased. This indicated longer average decay times. In another application, the use of fluorescence decay of Neodynium ion as an information carrier in a multimode fiber optic pressure sensor has been demonstrated (ref 2).

High quantum efficiency of the fluorescence, good mechanical and thermal properties, and weak temperature dependency of the quantum efficiency and decay rates are requirements for a good information carrier material. The rare earth ions incorporated into the various crystalline inorganic phosphors make these materials suitable as information carrier materials. Webster and Drickamer (ref 1) found that samples of doped phosphors with high concentrations of rare earth europium showed

emission characteristics that were quantitatively different from samples of lower concentrations when the pressure changed. Smaller changes in the emission characteristics were encountered with the higher concentration of doped europium phosphor samples. The total intensity of the spectral emission from these higher doped materials is also greater.

Relative intensity changes are determined rather than absolute number of photons recorded. The small shifts in peaks in the emission with pressure changes eliminated the need to correct the data for the response of the spectrometer to different wavelengths of light. It was also found from the spectral emissions of doped phosphors that they are temperature dependent as well (refs 3 and 4). For example, studies shown in figure 1 (ref 1) illustrate an increase in intensity with lower temperature. This may be explained as less energy is being delivered to the lower $^5D_2$ levels as $^5D_3$ level emission increases. Quenching through the charge transfer state makes less energy available to the lower levels. The data provide reproducibly measurable intensities with increasing temperatures and pressures. Based upon this information, the use of doped inorganic phosphors appeared promising as a remote sensor to monitor high pressures and temperatures.

EXPERIMENTAL TEST DESIGN

The experimental test setup is shown in figure 2; high-pressure optical cell in figure 3 (ref 5 and 6). The phosphor sample is surrounded by sodium chloride and sandwiched between two steel disks or hardened tool steel which move when an external force is applied to the pistons. The stepped NaC1 windows are pressed into the steel insert which is shrunk-fit into the steel body of the cell. The salt in the optical ports is supported by sapphire cylinders. This cell design can be used repeatedly to about 30 kbar without replacing steel disks and insert which tend to distort about 30 kbar.

An external force is applied to the sample in the cell in a press with a 10-ton hydraulic ram activated by a hand pump. The sample pressure is within 10% of that calculated from the mechanical advantage (203:1 ram to disk area) of the apparatus. The pressure scale, applicable to the recorded gauge reading for a particular disk set, is verified with the optical observation of the KC1 phase transition at 19.8 kbar. It is observed as a loss of transmittance when the salt undergoes the phase transformation (ref 5 and 6). Temperatures above ambient are achieved with four resistive heating elements placed in the steel body of the cell body and measured with a thermocouple.

A schematic of the apparatus used in this study is shown in figure 2. The phosphor sample is excited with 1 mW (1.1 mm diameter) of 325 nm radiation from a He-Cd laser.

The emission is viewed at right angles from the excitation axis through a 0.25-m monochrometer using a fl.2 50 mm camera lens. The signal is detected by a photomultiplier, amplified by an electrometer, and recorded on an X-Y recorder.

The material used was La_2O_2S:Eu with 1 mole percent europium doped in place of the lanthanum. The phosphor powder is mixed 1:10 with crushed NaCl crystals and pressed into a thin wafer in a pellet press. A rectangular section of this wafer is inserted into a slot previously cut into the NaC1 cylinder fused into the center of the high-pressure optical cell. This slot is milled at 45 degrees relative to the excitation/emission axis in order to detect front-surface luminescence. The sample pellet is carefully fuzed to the correct thickness and transparency. The sample and cell are now ready to be inserted in the high pressure station shown in figure 2.

DISCUSSION AND RESULTS

The emission spectrum of La_2O_2S:Eu recorded with the appartus is shown in figure 4 at ambient temperature. In the 450 to 659 nm spectral region, the emission lines from the $^5D_2$, $^5D_0$ excited states to the ^7F_i lower states are found. In the temperature range from ambient to about 50° C, the $Eu^{3+5}D_2$ lines in the shorter wavelength region are strongly temperature-sensitive, while the $^5D_1$ lines (toward the longer wavelengths) show a smaller sensitivity to temperature changes. For purposes of our detection limit and relative intensity scales, the 514-nm line ($^5D_2$-$^7F_3$) and the 555-nm line ($^5D_1$-$^7F_2$) were monitored. How these two lines are influenced by pressure at 35° C are shown in figure 5. The 514-nm line grows ten-fold at high pressures, while the 555-nm line is diminished by approximately 50%. This pressure response is interpreted as an inhibition of the $^5D_2$-charge transfer (CT) state transition probability and the consequent reduction in the $^5D_1$ population derived from the CT state. The 555-nm line is chosen to normalize the intensity, and the intensity ratios I_{514}/I_{555} are reported as a function of pressure at three temperatures. The ratio for three pressure runs at 35°C is shown in figure 6. The reproducibility for these types of measurements is excellent. It is also noted that pressure affects the intensity ratio only slightly below 10 kbar, but the increase in I_{514}/I_{555} accelerates steeply at the higher pressures. This behavior is consistent with the single-configurational model (refs 7 and 8). Here, the quenching process is activated, and the pressure-induced changes are consistant with an increase in energetic separation of the ground and charge transfer state. Specifically, if the change in activation energy with pressure is linear, then a linear dependence of $1nI_{514}/I_{555}$

versus P is anticipated from the model (ref 1). Such a plot closely approximates the linearity over this pressure and temperature interval (fig. 1). Note that the temperature offset of 27°C corresponds to about 9.5 kbar in pressure.

CONCLUSION AND RECOMMENDATIONS

The La_2O_2S:Eu phosphor is an excellent indicator of pressure in the 20 to 50°C temperature range. TGhe I_{514}/I_{555} value obserrved in an unknown environemnt can be related to thepressure if the temperature is independently known. Additional experiments are obviously needed to separate the two environmental effects. According to our present knowledge of these systems, nonradiative transitions to neighboring charge transfer bands would be influenced by both P and T. It is required that simultaneous measurements of at least two phosphors with known and distinct P-T offsets be made. A systematic photophysical study of other luminescent materials in which the P and T quenching mechanisms are uncoupled is needed to determine if the material is either insensitive to pressure or temperature. For example, f-f transitions far removed from charge transfer bands may be shielded from pressure perturbations. However, temperature changes are reflected in their emission from the redistribution of sublevel populations. Similarly, a search for a luminescent material whose quantum efficiency is T-independent by P-dependent would be of great value for pressure and temperature monitoring applications. These studies can be refined by changing the excitation wavelengths of luminescent materials and investigating the influence of thermal traps and mechanical defects in the solid materials. Clearly, the continued investigation of the photophyhsical behavior of phosphors in extreme environments is of fundamental significance and practical importance in P-T measurements.

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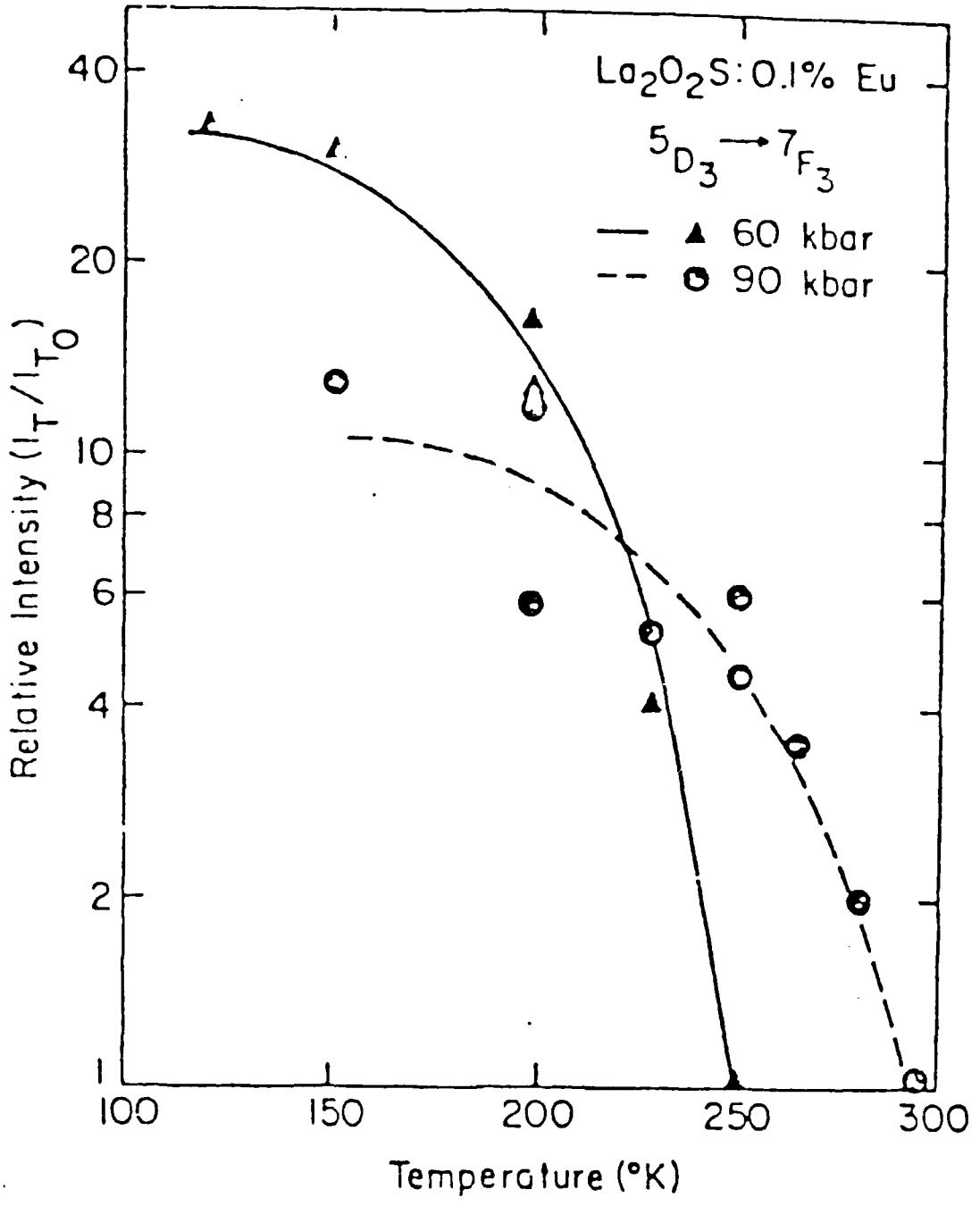


Figure 1. Intensity increase with lowering temperature

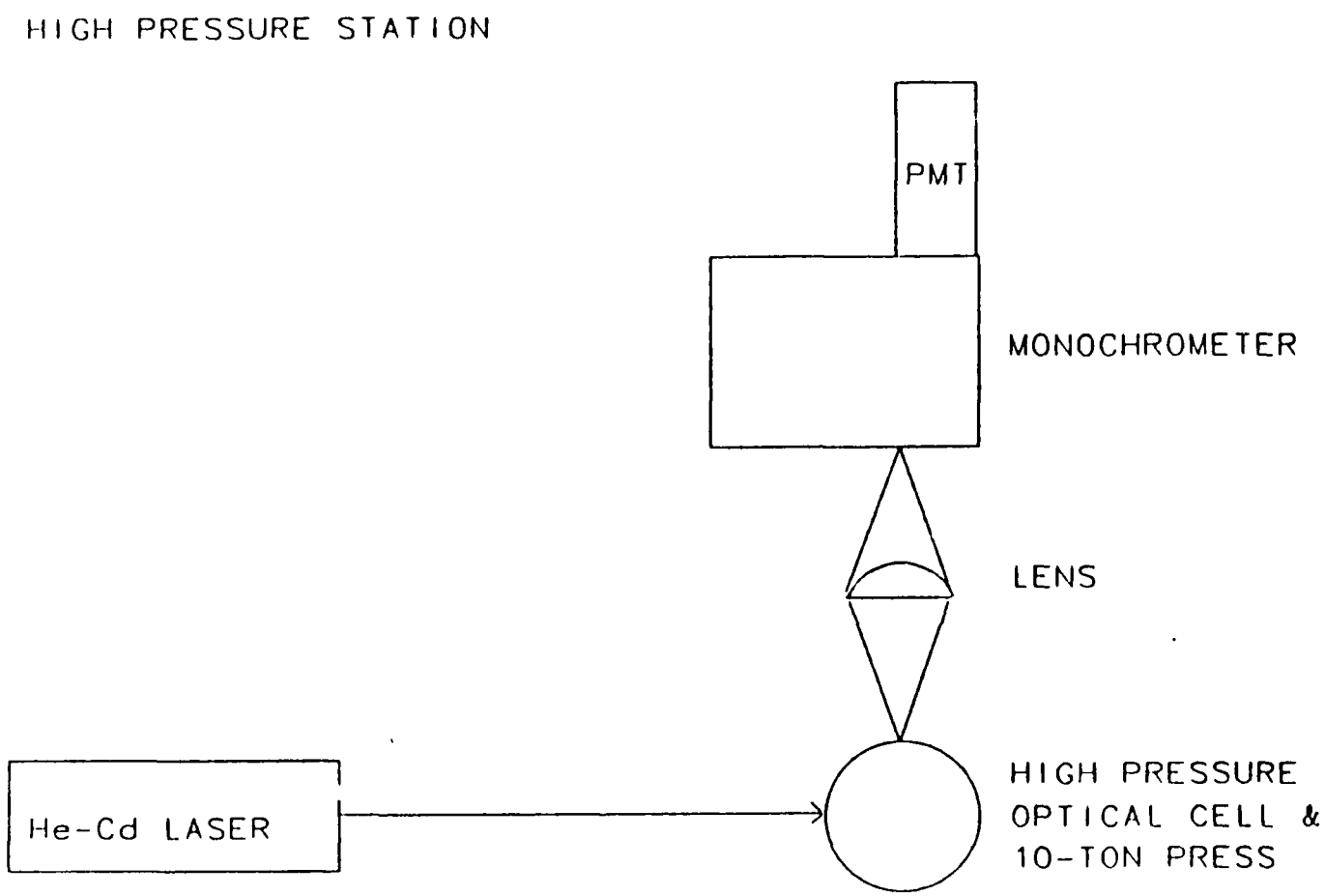
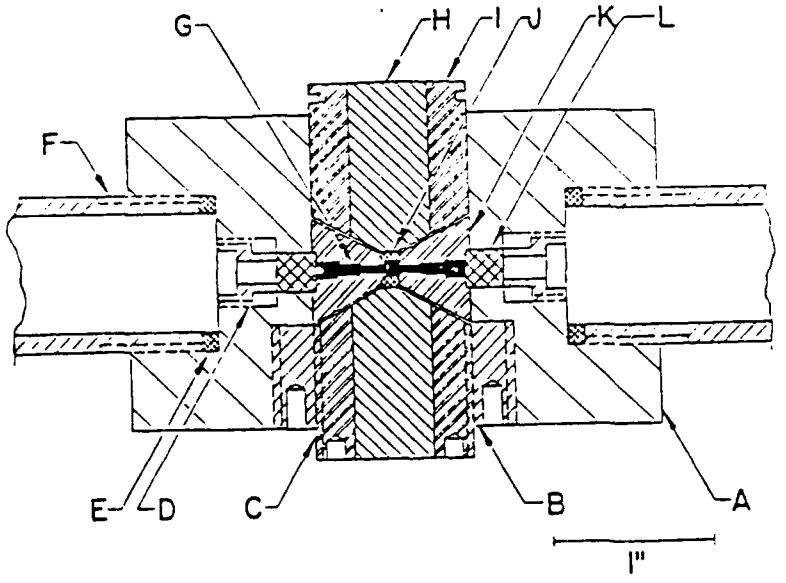
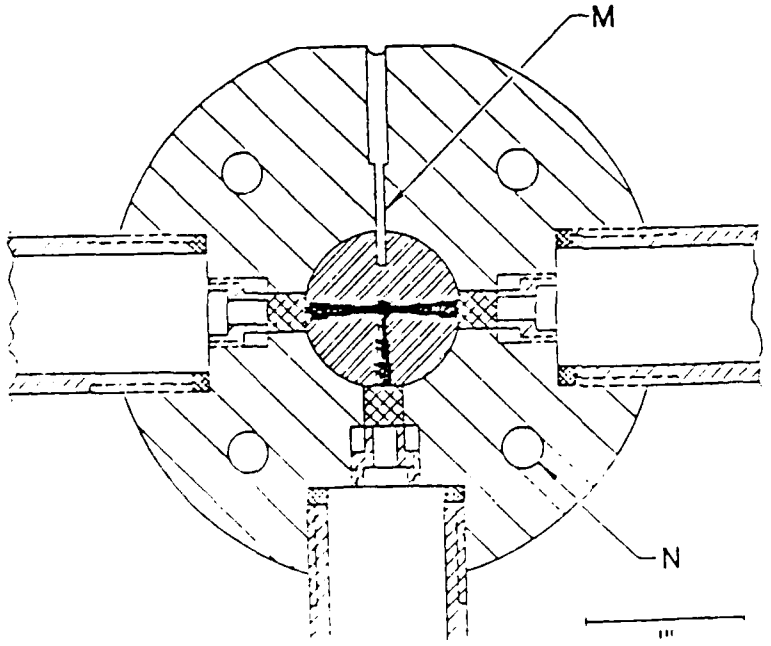


Figure 2. Schematic of high-pressure setup



side view



top view

A - outer tool steel jacket

B - brass piston guide

C,I - piston sleeve

D - brass retainer for sapphire window

E - Teflon gasket

F - window vacuum tubes

G - three-stepped window parts filled with NaC1 in tool insert

H - tungsten cartridge piston

J - sample pellet located between these two steel disks

K - tool insert

L - sapphire window

Figure 3. Cross-section of high pressure optical cell

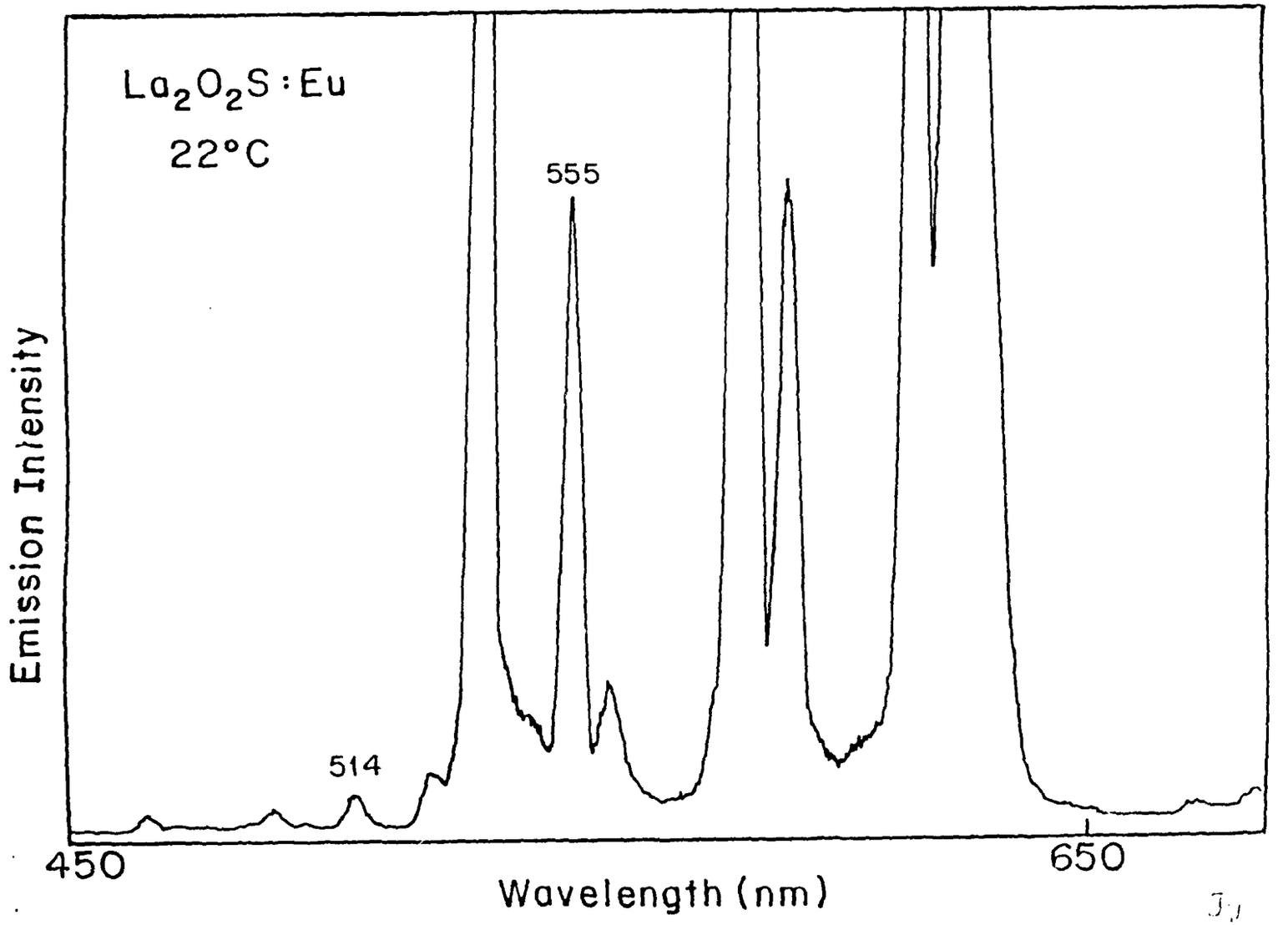


Figure 4. Emission spetrum of La_2O_2S;Eu under ambient conditions with 325 nm excitation

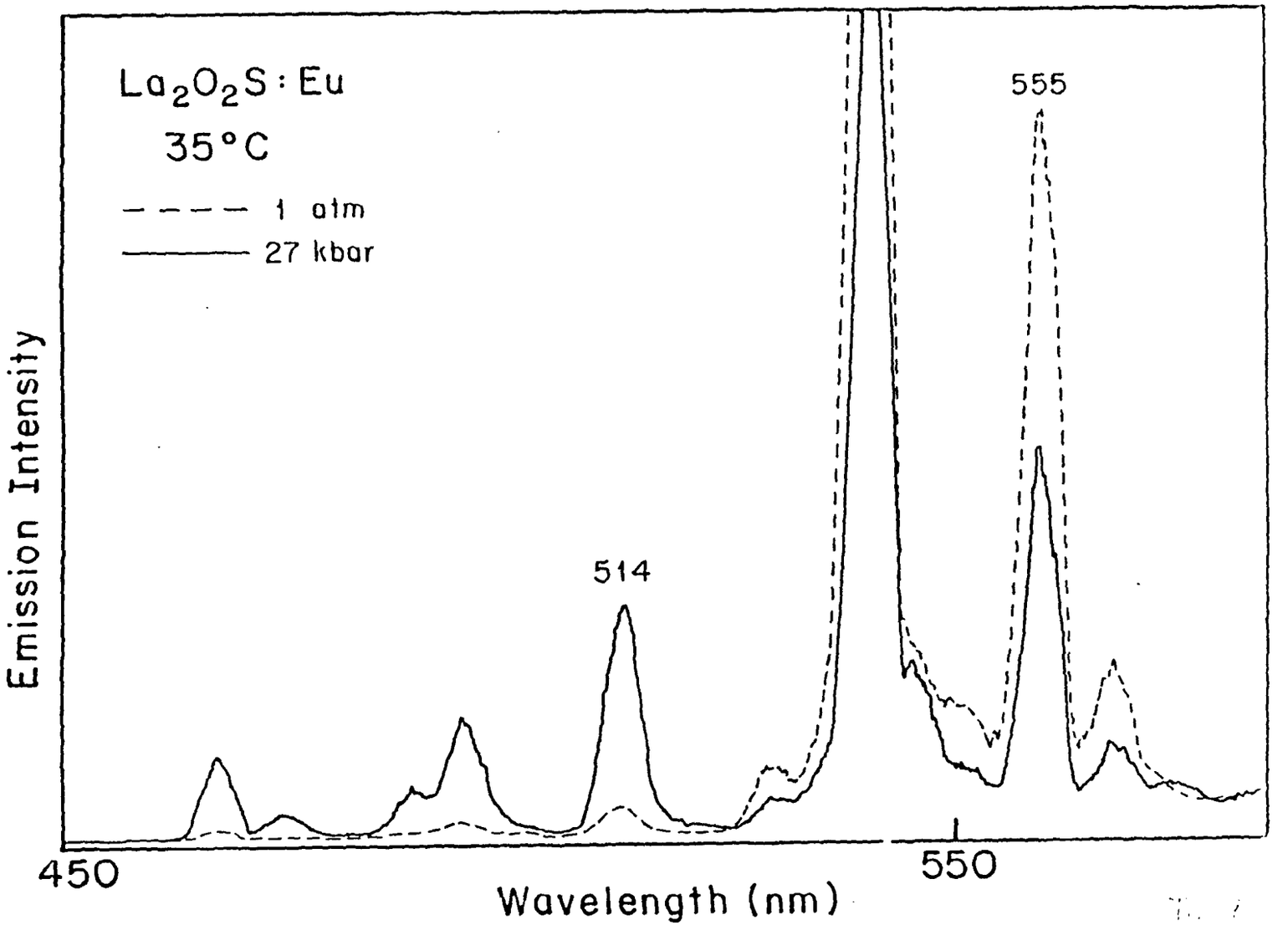
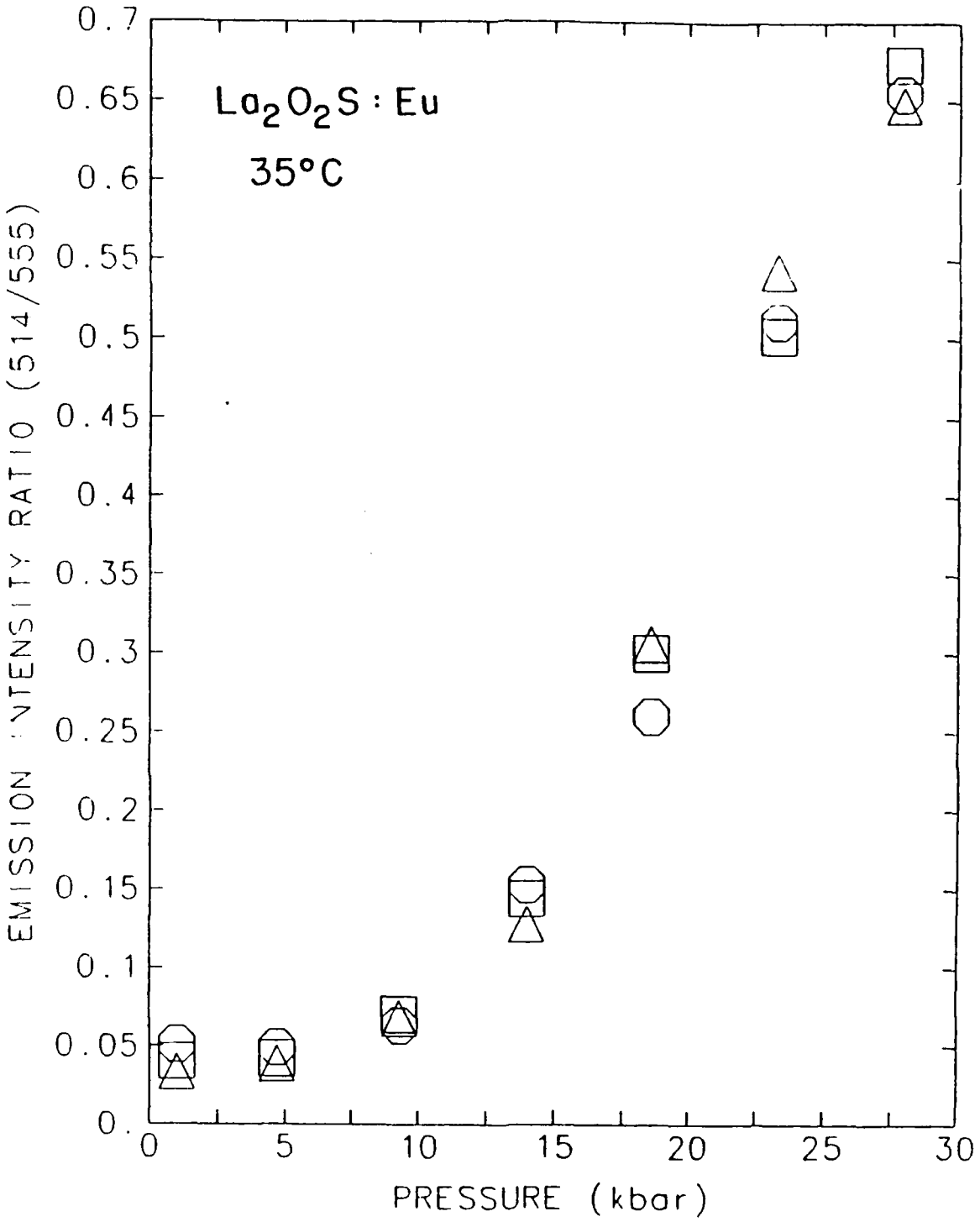


Figure 5. Emission spectrum of La_2O_2S:Eu at 1 atm and 27 kbar



NOTE: Three separate runs, (△, □, ⬡)

Figure 6. Pressure dependency of the emission intensity ratio at 35°C

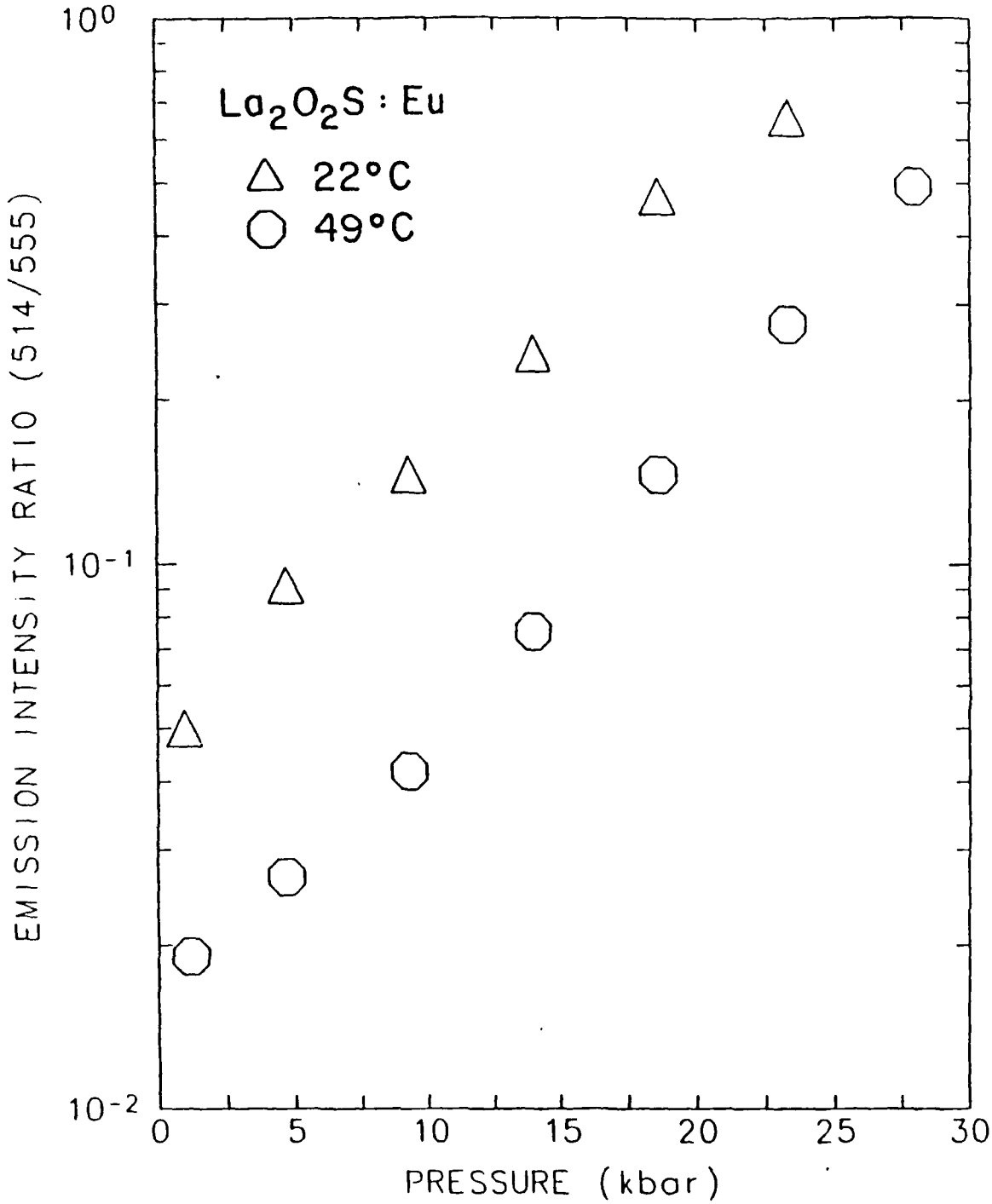


Figure 7. Logarithmic dependence of the emission intensity ratio with pressure at 22 and 49°C

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